

NACA TN 3107 9076



NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

TECHNICAL NOTE 3107

EFFECT OF HEAT TREATMENT UPON THE MICROSTRUCTURE
AND HARDNESS OF A WROUGHT COBALT-BASE ALLOY

STELLITE 21 (AMS 5385)

By F. J. Clauss and J. W. Weeton

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Cleveland, Ohio



Washington

March 1954

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WROUGHT COBALT-BASE ALLOY, STELLITE 21 (AMS 5385)

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SUMMARY

The response of wrought Stellite 21, a typical cobalt-base alloy, to heat treatment was investigated. The alloy was solution-treated and transformed both by aging and isothermal transformation at temperatures of 1200° to 1950° F for periods of 5 minutes to 72 hours. Metallographic studies and hardness measurements were made to relate the manner and extent of precipitation with time, temperature, and type of heat treatment. The results presented herein may be considered preliminary to a more extensive study of relations of microstructures to the high-temperature properties of wrought Stellite 21.

A period of 72 hours at 2250° F was sufficient for effective solution-treatment of the material so that little or no residual precipitate was visible. Pearlitic structures were formed at grain boundaries of solution-treated specimens after isothermal transformation at 1950°, 1750°, and 1500° F. In addition to pearlite, a star-shaped Widmanstaetten structure occurred in specimens transformed at 1750° and 1500° F. Little visible precipitate was formed by isothermal transformation at 1200° F for times up to 72 hours. The aging treatments resulted in the formation of scattered precipitates along slip lines and twin boundaries. Spheroidization and agglomeration of precipitates occurred at temperatures as low as 1500° F and were initiated more rapidly in aged specimens than in isothermally transformed specimens.

The hardness of the solution-treated specimens was Rockwell C-20 to C-22. Isothermal or aging treatments above 1200° F appreciably increased the hardness of specimens. A maximum hardness of Rockwell C-42 was developed by aging 72 hours at the temperature range from 1400° to 1500° F. Overaging or softening occurred within 72 hours at temperatures of 1750° F and above for aging, and 1950° F for isothermal transformation. The specimens aged or isothermally transformed at 1200° F did not increase in hardness. Specimens aged at 1500° F and those isothermally transformed at 1750° and 1500° F continued to increase in hardness up to 72 hours at respective temperatures.

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INTRODUCTION

The investigation reported herein was undertaken as part of a fundamental program concerned with the general metallurgical improvement of turbine-blade alloys to provide a basis for proper selection and heat treatment of alloy materials. Previous work (refs. 1 to 3) had demonstrated that improvement in the life of turbosupercharger blades of Stellite 21 could be obtained by suitable heat treatment. For example, it was found that a heat treatment which produced a lamellar type of precipitation in the microstructure, resembling pearlite in steel (ref. 3), approximately doubled the average blade life. (Pearlite, in this report, refers to the lamellar-type structure.)

Wrought Stellite 21 was used in the present study rather than the cast alloy in an effort to avoid grain-size variables and to permit a more thorough homogenization by solution treatment. X-ray diffraction studies of this alloy (ref. 4) showed that the matrix phases of the as-wrought structure consisted of both face-centered cubic and hexagonal close-packed structures and that the Cr_{23}C_6 -type carbide was the predominant minor constituent. Furthermore, it was shown that the wrought structure responded well to solution treatment; upon cooling to various isothermal transformation levels, the alloy formed varying quantities of minor phases, principally in the form of pearlite.

Stellite 21, in several conditions of heat treatment, and several other cobalt-base alloys have been investigated metallographically and by X-ray diffraction methods (refs. 1 to 11). The carbides Cr_7C_3 , Cr_{23}C_6 , M_6C (where M may be any carbide-forming element), and the sigma phase have been the principal minor phases detected in this alloy. The grain-boundary precipitants, part of the pearlitic structure, and many of the massive carbides which are usually observed in Stellite 21 were largely Cr_{23}C_6 (refs. 4, 5, 8, 10, and 11). The matrix of Stellite 21 after solution treatment has been shown to be face-centered cubic and this structure is known to transform to the hexagonal close-packed structure at temperatures of 1500° to 1950° F, (refs. 4 to 6). In pure cobalt, transformation of the face-centered cubic to the hexagonal close-packed structure was shown to occur by a martensitic type of reaction (ref. 12), and this type of reaction has been assumed to apply to the similar transformation in the cobalt-rich solid solution which makes up the matrix of Stellite 21.

In the references previously cited, no attempts were made to relate the quantities of precipitation with the variables of time or temperature of isothermal or aging transformations. In the present investigation, a systematic study of the microstructural and hardness changes produced in wrought Stellite 21 by heat treatment has therefore been made in order to relate the amount and type of precipitation with the time, temperature, and manner of heat treatment. The results to be presented are preliminary to a more extensive study of relations of microstructures to the high-temperature properties of Stellite 21.

PROCEDURE

Wrought Stellite 21 of the following chemical analysis was employed in the investigation reported herein:

C	Cr	Ni	Co	Mo	Fe	Si	Mn
0.29	28.75	3.01	Bal.	5.52	0.33	0.44	0.56
(61.10)							

The material was fine-grained, with an A.S.T.M. grain size of about 8. The microstructure of the as-wrought material is shown in figure 1.

Slugs of this material were cut from 3/4-inch-diameter bar stock supplied by the manufacturer and heat-treated in electric furnaces using Globar heating elements. The metal slugs were placed inside a 2¹/₄-inch-
inside-diameter tube of Inconel X in the heating zone of the furnace. Temperatures, which were measured by a platinum - platinum plus 13 percent rhodium thermocouple located immediately above the samples, were controlled to $\pm 10^{\circ}$ F of the nominal temperatures. Argon was circulated through the tube to provide an inert atmosphere during heat treatment. The gas was originally 99.6 percent pure argon and was further purified before entering the specimen tube by passing over shavings of titanium metal at 1300^o to 1400^o F.

The metal slugs were placed in the furnace at a temperature of approximately 2000^o F and held at this temperature for 1 to 2 hours. The purpose of holding at this temperature was to effect some preliminary homogenization of the structure and to minimize any tendencies towards eutectic melting at higher temperatures. The temperature was then raised to a solution-treating temperature of 2250^o F and held for 72 hours to produce a homogeneous solid solution of carbon in the face-centered cubic matrix of the alloying metals.

At the end of the solution treatments, the specimens were either water-quenched and aged or transferred directly to furnaces held at lower temperatures for isothermal transformations. The nominal transformation temperatures were 1200^o, 1500^o, 1750^o, and 1950^o F, and times at each of these temperatures were 5 minutes and 2, 4, 25, and 72 hours. In order to obtain sufficient data for drawing representative hardening curves, additional heat treatments were carried out at intermediate temperatures and times, as indicated subsequently. All specimens were water-quenched after the transformation treatments.

The use of salt baths for the isothermal transformation treatments was found impractical because of the severe corrosion of the specimens, especially at high temperatures and long transformation times.

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Rockwell hardness measurements were made on the heat-treated specimens after outer surfaces had been removed by sanding or grinding. A minimum of three hardness measurements was made on each specimen. The specimens were then mounted, polished, and etched for metallographic study, and 5-percent aqua regia was used electrolytically as the etchant.

RESULTS

Solution-treated structure. - The solution treatment was sufficiently complete to produce a solid solution showing little or no residual precipitate in the microstructure, as shown in figure 2. While considerable grain growth occurred during solution treating, the final grain size was still less than that of cast Stellite 21. Little further grain growth was evident during subsequent aging or isothermal transformation.

Precipitation by isothermal transformation. - Isothermal transformation of the solution-treated material at a temperature of 1950° F resulted in the formation of a lamellar, pearlitic type of structure along the grain boundaries as shown in figure 3(a). Pearlitic precipitation has started in less than 5 minutes and appears to be complete after 2 hours at 1950° F. "Divorcing" of the pearlite, or the tendency of the lamellae to break up and agglomerate into small spheroidized particles, is evident as the time of transformation increases, which is analogous to the behavior of steels.

Isothermal transformation at 1750° F (fig. 3(b)) was largely by pearlitic formation along the grain boundaries, as at 1950° F, with some scattered precipitation within the grains. The rate of pearlitic growth was less at 1750° F than at 1950° F, and the interlamellar distance in the pearlite was reduced by the lowering of the temperature. There was an increase in the amount of precipitate for times up to 72 hours. Spheroidization can be noted in the microstructure after 25 and 72 hours.

Unlike specimens transformed at 1750° and 1950° F, those transformed at 1500° F have only a small amount of pearlite (fig. 3(c)). The precipitate formed at this temperature was almost entirely of a star-shape (Widmanstaetten type) distributed throughout the grains. The amount of visible precipitate increases with the time of transformation up to 72 hours. The photomicrographs at X100 (fig. 3(c)) show a distinct layer of precipitate-free matrix along the grain boundaries. As in other alloys, this effect is probably the result of the agglomeration of precipitate in the grain boundaries (ref. 13). The few small patches of pearlite appearing at some grain boundaries consist of very closely packed lamellae, unresolved at X750 magnification.

At 1200° F, little visible precipitate was formed by isothermal transformation for periods up to 72 hours (fig. 3(d)). Traces of pearlite were observed in specimens transformed for 25 and 72 hours, although this may not be apparent in the photomicrographs.

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Precipitation by aging. - In sharp contrast to the precipitate formed by isothermal transformation, no pearlite was formed by any of the aging treatments. Aging at temperatures of 1950°, 1750°, and 1500° F resulted in precipitation almost entirely along slip lines and twin boundaries through the grains as shown in figures 4(a), 4(b), and 4(c). These slip lines and twin boundaries are the result of thermal stresses set by water quenching from the solution-treating temperature prior to aging. Precipitation by aging was very rapid at 1950° and 1750° F and appeared to be complete in 1 to 2 hours. Continued aging at these temperatures resulted in the agglomeration or coalescence of the precipitate particles.

The amount of visible precipitate formed by aging at 1500° F continued to increase up to 72 hours.

Short-time aging at 1200° F (fig. 4(d)) gave no visible precipitate, while longer times (25 to 72 hr) at 1200° F developed a small amount of precipitate along slip lines.

Grain boundary behavior. - Agglomeration of the precipitates in the grain boundaries occurred with increasing time. This was particularly evident at the higher temperatures of isothermal transformation, and could be observed down to 1500° F. At 1200° F the grain boundaries remained fine and continuous with increasing time of aging or isothermal transformation.

For specimens aged at 1200° F, the grain boundaries were very fine and difficult to show clearly by the etching technique. In the case of the specimen aged 5 minutes, it was necessary to substitute a more corrosive etchant (1:1 HCl, electrolytically) than 5-percent aqua regia in order to attack the grain boundaries, since the specimen was still essentially in the solution-treated condition.

Hardness. - The hardness of the specimens after solution treatment lay in the range from C-20 to C-24. Subsequent hardening during aging or isothermal transformation at temperatures below the solution-treating range is shown in figures 5 to 8. These figures present the data in both the conventional type of hardening curve (hardness against the logarithm of time of aging or isothermal transformation at constant temperature, figs. 5 and 6) and in cross plots to show the effect of temperature (hardness against temperature at constant time, figs. 7 and 8). In constructing these hardness curves, the experimental points were first plotted on both types of figure. Trial curves were next drawn to "average out" the experimental deviations on each separate type of plot. Values were read from the trial curves of one type of figure and cross-plotted on the other. New curves were then drawn to best approximate both the experimental points and the values obtained from the trial curves. The process was repeated until the final curves on both sets of figures were consistent and gave the best representation of the general trend within the limits of experimental error.

At 1950° F, both aged and isothermally transformed specimens hardened to a maximum hardness of about Rockwell C-33 in a matter of several minutes, after which some overaging and softening took place. This softening is related to the agglomeration or coalescence of the precipitate in the microstructure, as noted previously.

At 1750° F, the aged specimens hardened more rapidly than those isothermally transformed, reaching a maximum hardness of Rockwell C-36 after 2 hours of aging. Softening of the aged specimens after 2 hours was presumably due to the coalescence of the fine carbides precipitated along the slip lines. The isothermally transformed specimens continued to harden to 72 hours, when their hardness was Rockwell C-38.

At 1500° F, the aged specimens again hardened more rapidly than those isothermally transformed, but there was no evidence of overaging or softening within the time investigated. Instead there was a steady increase in hardness with time for both types of heat treatment. Aging for 72 hours at 1500° F gave a hardness of Rockwell C-42, while isothermal transformation for the same time resulted in a hardness of Rockwell C-39. Little further increase in hardness due to aging could be obtained by changing the temperature of aging, and 1500° F appeared to be the temperature for maximum hardness.

At 1200° F, there was no measurable increase in hardness with the time of aging or isothermal transformation, and the hardness was constant at about Rockwell C-24 for the aged specimens and Rockwell C-22 for the isothermally transformed specimens. This is as would be expected from the microstructural behavior at this temperature, which showed little change in microstructure from that of the solution-treated condition.

DISCUSSION

After a 72-hour solution treatment at 2250° F, wrought Stellite 21 is essentially a homogeneous solid solution. This has been shown to have a face-centered cubic structure (refs. 4, 10, and 11). Subsequent transformation at lower temperatures occurs both with changes in the crystal structure of the matrix and with the formation of minor phases. The matrix transformation from face-centered cubic (alpha phase) to hexagonal close-packed (beta phase) structures has already been described as taking place by a martensitic type of reaction (refs. 9 to 11). The formation of the minor phases, principally carbides with perhaps some sigma phase, may occur by precipitation directly from a single-phase solid solution, by a eutectoid decomposition, and/or by the transformation from one type of carbide to a second type; in any case, a nucleation and growth involving diffusion of atoms through the lattice is the most likely mechanism as discussed in the following paragraphs.

2809 Carbide precipitation is nucleated almost entirely at grain boundaries in the case of the specimens isothermally transformed at 1750° and 1950° F. Subsequent growth to form pearlite occurs with the diffusion of carbon from within the grains to the grain boundary sites where it reacts with the carbide-forming elements to form the carbide lamellae (largely Cr_{23}C_6 with some of the chromium displaced by molybdenum or other carbide-forming elements). This results in a localized depletion of chromium in the solid-solution lamellae alternating with the carbide lamellae and in adjacent areas, which therefore have less resistance to attack by the etchant. The depleted zones appear to have a definite and distinct border between the solid-solution lamellae in the pearlite and the matrix in the main body of the grains (fig. 9), as has been noted previously with other lamellar precipitates (ref. 13). A similar observation was made by DeVries and Mohling (ref. 14) in the case of a high-temperature alloy having the following composition:

C	Cr	Co	Ni	Si	Mn	Mo	W	Fe
0.51	20.02	27.76	19.08	0.40	0.86	2.11	6.83	22.26

Slightly lighter areas were observed to surround the carbide precipitates in the microstructure of this alloy, and it was felt by the authors that this was due to concentration gradients rather than to a fully developed second phase. These areas were reasoned to be areas impoverished of austenite strengtheners by the formation of the precipitates and thereby weakened, which would account for the lower stress-rupture properties obtained for this alloy as compared with other alloys studied by these investigators. No direct proof is offered for the correctness of this view, and others (ref. 15) have explained the same effect as being due to the difference in orientation between the two matrix areas brought about by recrystallization. A third possibility is that the differently etched areas are due to the presence of beta phase in the lamellae as distinct from the alpha phase of the remaining matrix (ref. 4).

The starlike Widmanstaetten structure of the carbide particles which appear as the main precipitate formed by isothermal transformation at 1500° F strongly suggests that this type of precipitate forms on the (111) octahedral planes of the face-centered cubic matrix. This type of precipitate also occurs by isothermal transformation at 1750° F, where it forms a minor part of the total.

Precipitation is, in general, favored on those crystallographic planes of the parent matrix on which the atomic spacing is nearly equal to that on the adjoining plane of the precipitant. This condition is readily shown for the precipitation of the Cr_{23}C_6 type of carbide on the (111) plane of the face-centered cubic matrix; thus, if a value of 3.566 Å is used for the lattice parameter of face-centered cubic Stellite 21, which is the value reported by Rosenbaum (ref. 16), the distance between

the metal atoms on the (111) plane is calculated to be $0.707 \times 3.566 = 2.52$ A. The distance of closest approach between the metal atoms for a carbide of the type $(\text{Cr,Fe})_{23}\text{C}_6$ is given by Goldschmidt (ref. 17) as 2.45 to 2.51 A. This is a satisfactorily close match to favor precipitation of Cr_{23}C_6 in the suggested manner. It might also be noted that the close structural relation between this carbide and the austenitic matrix makes it possible that, in its initial stages of formation, the carbide forms from the austenite by a spontaneous transformation of the lattice, not involving atomic diffusion, as Goldschmidt points out.

In the case of the aged specimens, carbide precipitation is almost entirely along slip lines, or twin boundaries, with little or no tendency towards pearlitic or Widmanstaetten type formations. Nucleation sites for this type of precipitation are provided by thermal stresses set up by water quenching from the solution-treating temperature prior to aging. These slip planes are the (111) planes in the face-centered cubic matrix.

The hardening behavior of Stellite 21 may be termed normal. Initial increases in hardness upon aging specimens at temperatures above 1200°F may be observed. Prolonged aging or heat treating at 1750° and 1950°F causes the hardness to decrease after relatively short times. The softening that then occurs may be associated with spheroidization and agglomeration of the precipitate. The initial rate of hardening is greater for specimens transformed by aging than for those isothermally transformed at the same temperature, as would be expected from the greater number of nucleation sites provided by water quenching in the first case.

SUMMARY OF RESULTS

Investigation of the effects of time, temperature, and manner of heat treatment on the amount and type of precipitation in wrought Stellite 21 has given the following results:

1. Solution treatment of 72 hours at 2250°F was sufficient to produce a solid solution showing few or no residual precipitates in the microstructure. Grain growth occurred during solution treatment at 2250°F , and after 72 hours the grain size increased from A.S.T.M. 8 (as wrought) to a mixed grain size with some grains 10 to 20 times larger than A.S.T.M. 1.

2. Isothermal transformation of the solution-treated material at temperatures of 1950° , 1750° , and 1500°F resulted in the formation of pearlite along grain boundaries. Both the quantities of pearlite and the interlamellar spacing decreased with decreasing temperature.

3. Widmanstaetten structures formed by isothermal transformation at temperatures as high as 1750°F . At 1500°F , Widmanstaetten precipitation predominated over pearlitic precipitation.

4. Little visible precipitate was formed by isothermal transformation at 1200° F for times up to 72 hours.

5. Aging the solution-treated and water-quenched material at temperatures from 1200° to 1950° F resulted in precipitation principally along slip lines and twin boundaries.

6. With sufficiently prolonged times of aging or isothermal transformation, spheroidization and agglomeration of the precipitated carbide particles occurred at temperatures as low as 1500° F. This occurred more rapidly in the structures formed by aging than in those formed by isothermal transformation.

7. The hardness of the material after solution treatment for 72 hours at 2250° F and water quenching was Rockwell C-20 to C-24. The temperature for developing maximum hardness in the solution-treated material was between 1400° and 1500° F. Aging for 72 hours in this temperature range produced a hardness of Rockwell C-42. The maximum hardness for isothermal transformation was about Rockwell C-40 and was developed between 1500° and 1600° F.

8. Hardness was not increased by aging or isothermal transformation at 1200° F. Specimens aged at 1500° F and those isothermally transformed at 1500° and 1750° F continued to harden up to 72 hours. Overaging or softening occurred within 72 hours at temperatures of 1750° and above for aging and at 1950° F for isothermal transformation.

9. Aging the solution-treated and water-quenched material generally resulted in a more rapid increase in hardness than isothermal transformation at the same temperature.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, September 4, 1953.

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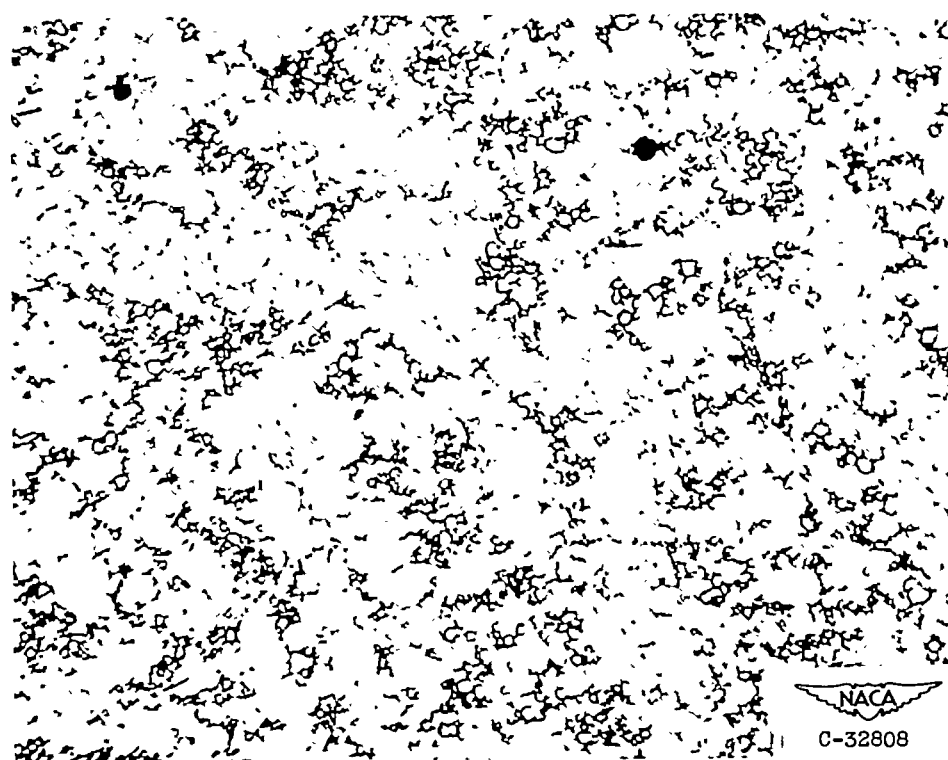


Figure 1. - Microstructure of wrought Stellite 21 in as-wrought condition. Electrolytically etched in 5 percent aqua regia. X750.

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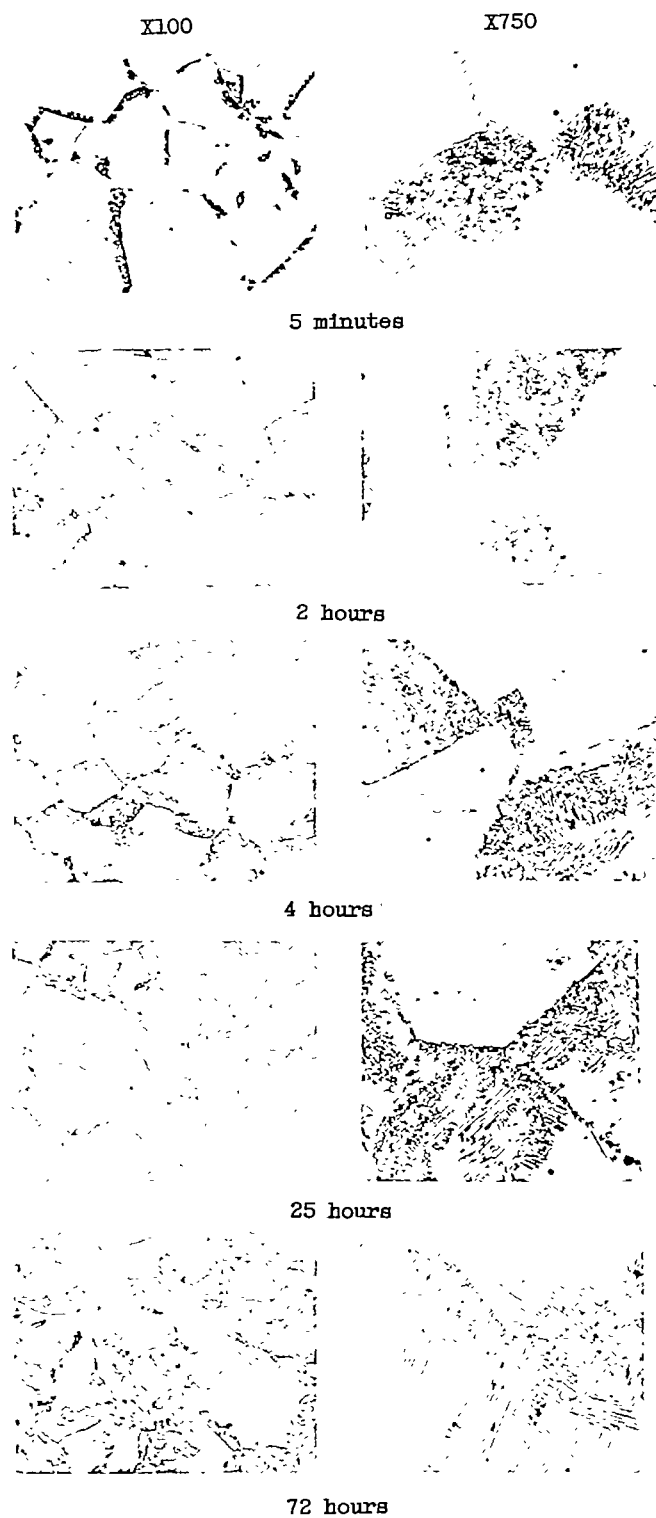
X100.



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X750.

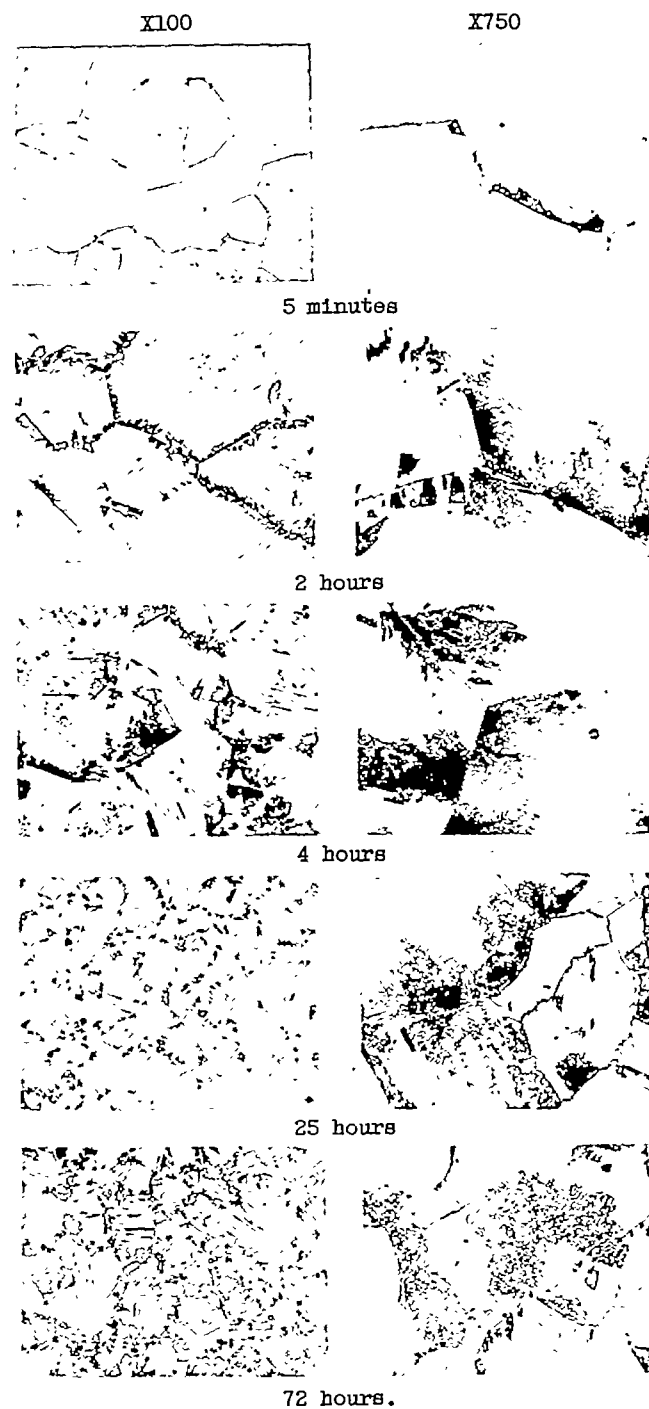
Figure 2. - Microstructure of wrought Stellite 21 solution-treated 72 hours at 2250° F and water-quenched. Electrolytically etched in 5-percent aqua regia.



(a) Temperature of transformation, 1950° F.

Figure 3. - Microstructure of Stellite 21 after isothermal transformation.

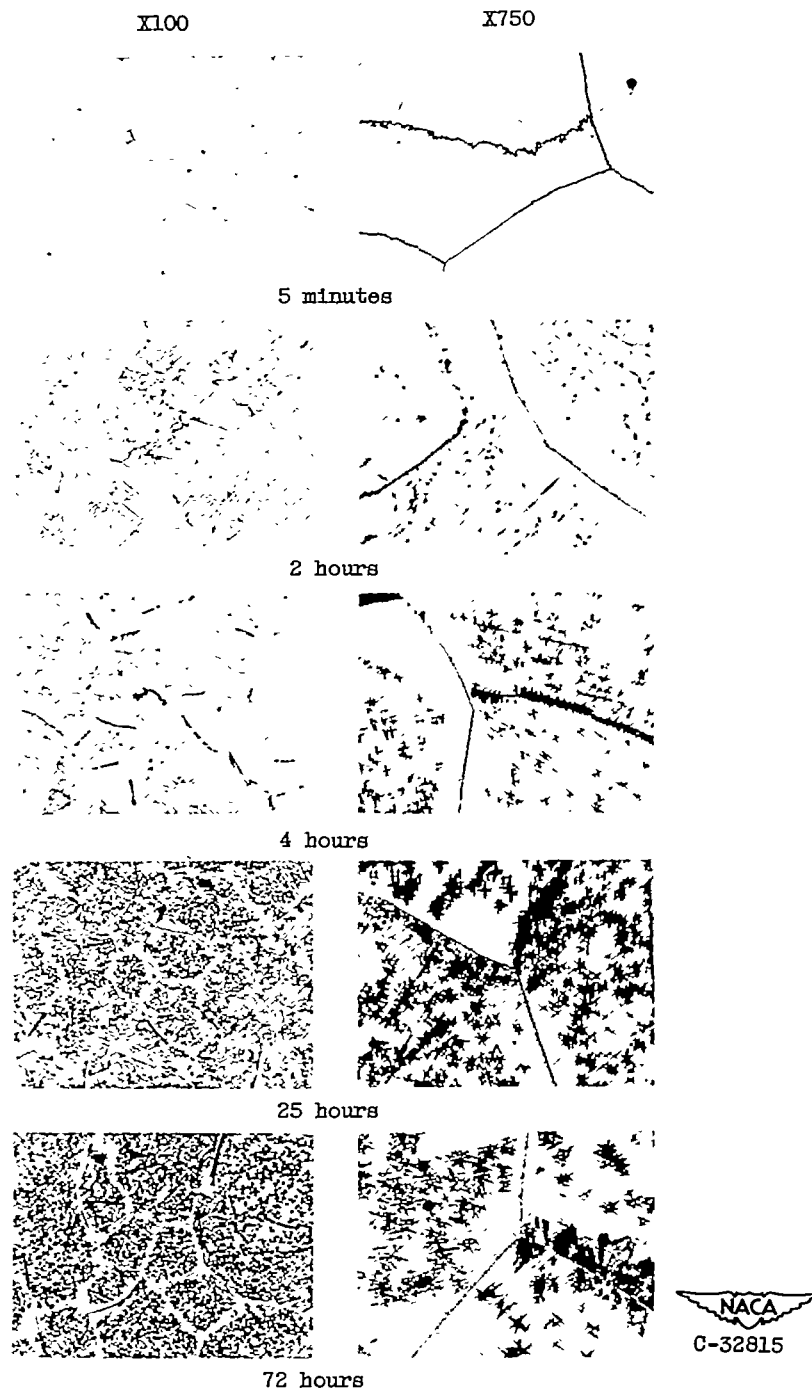
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(b) Temperature of transformation, 1750° F.

Figure 3. - Continued. Microstructure of Stellite 21 after isothermal transformation.



(c) Temperature of transformation, 1500° F.

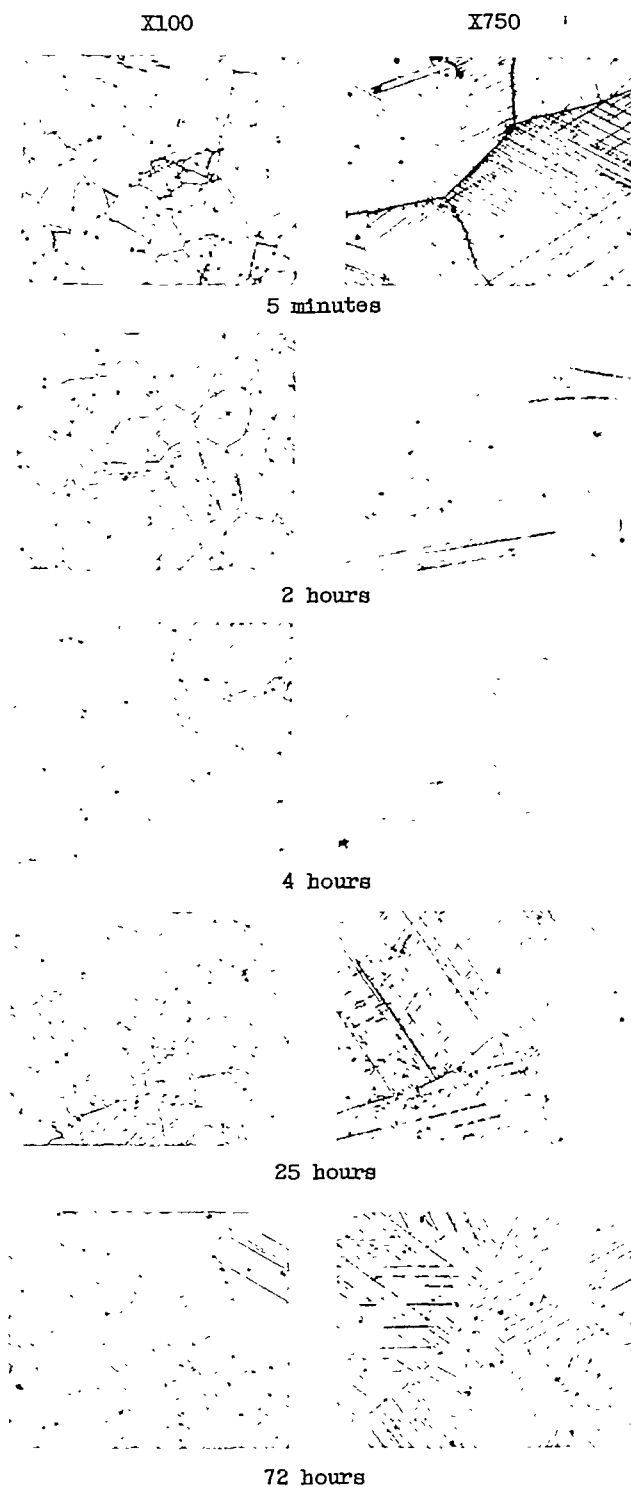
Figure 3. - Continued. Microstructure of Stellite 21 after isothermal transformation.

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(d) Temperature of transformation, 1200° F.

Figure 3. - Concluded. Microstructure of Stellite 21 after isothermal transformation.

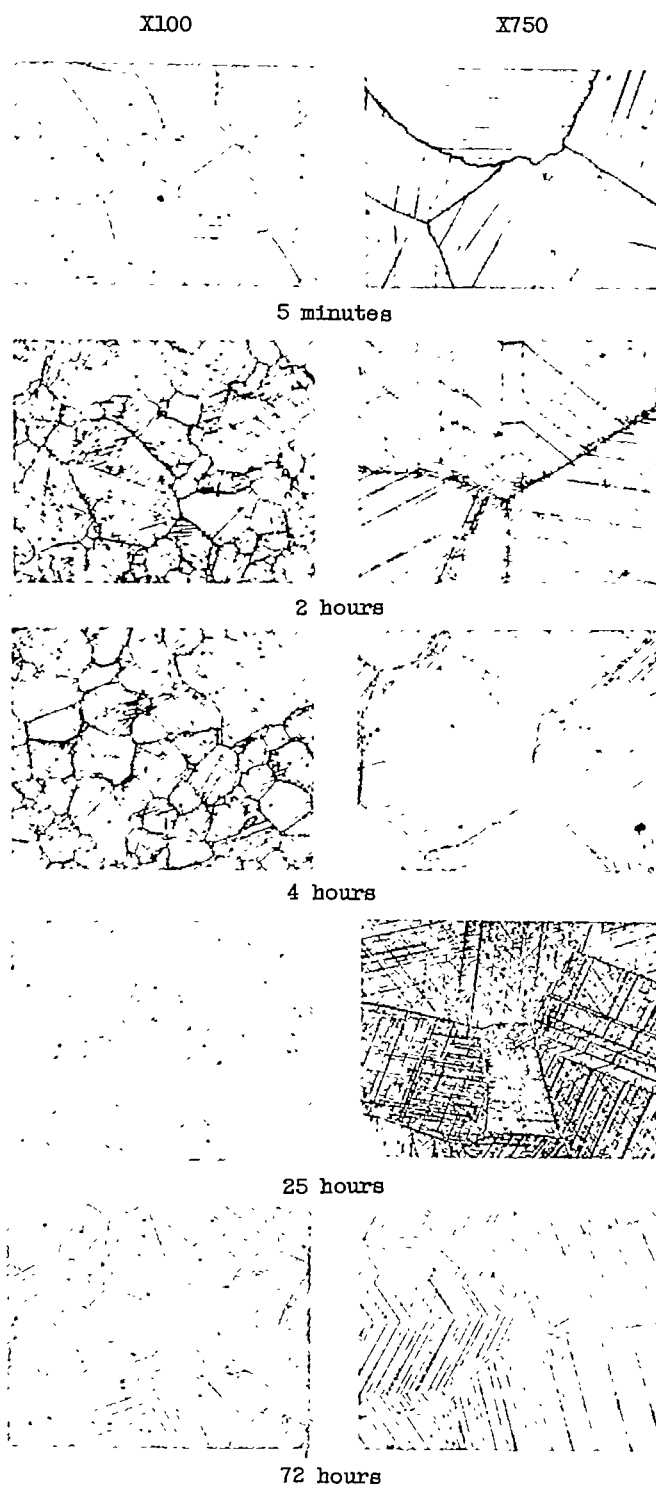


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(a) Aged at 1950° F.

Figure 4. - Microstructure of Stellite 21 after aging.

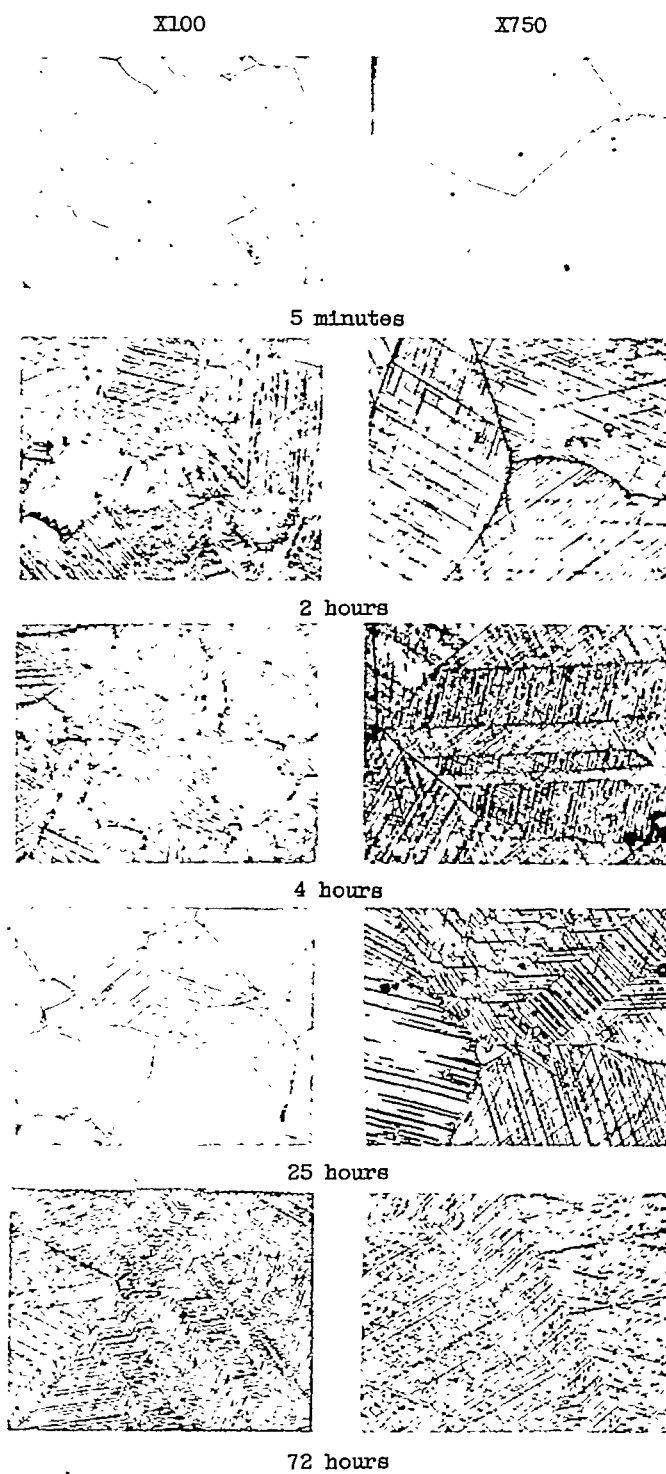
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(b) Aged at 1750° F.

Figure 4: - Continued. Microstructure of Stellite 21 after aging.



(c) Aged at 1500° F.

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Figure 4. - Continued. Microstructure of Stellite 21 after aging.

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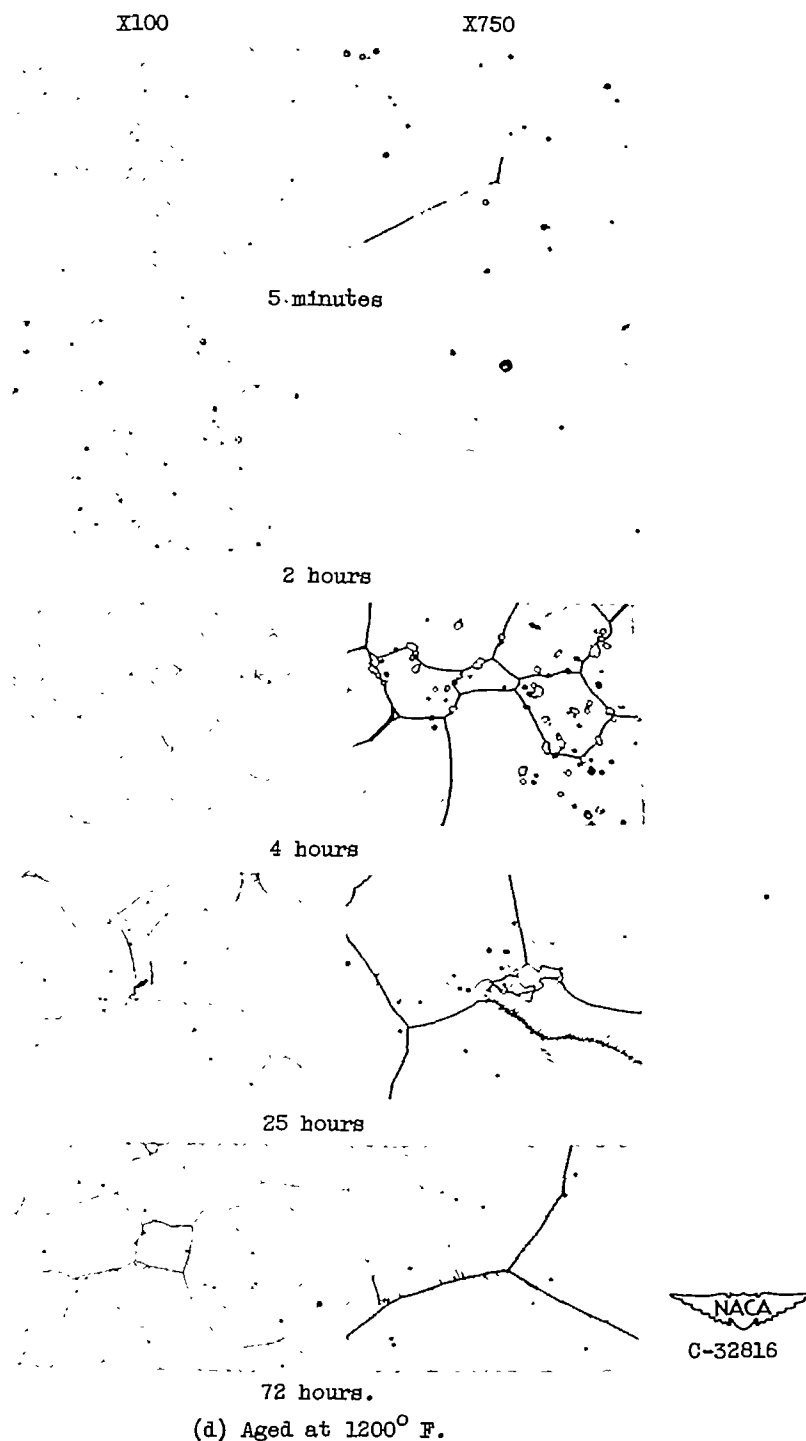


Figure 4. - Concluded. Microstructure of Stellite 21 after aging.

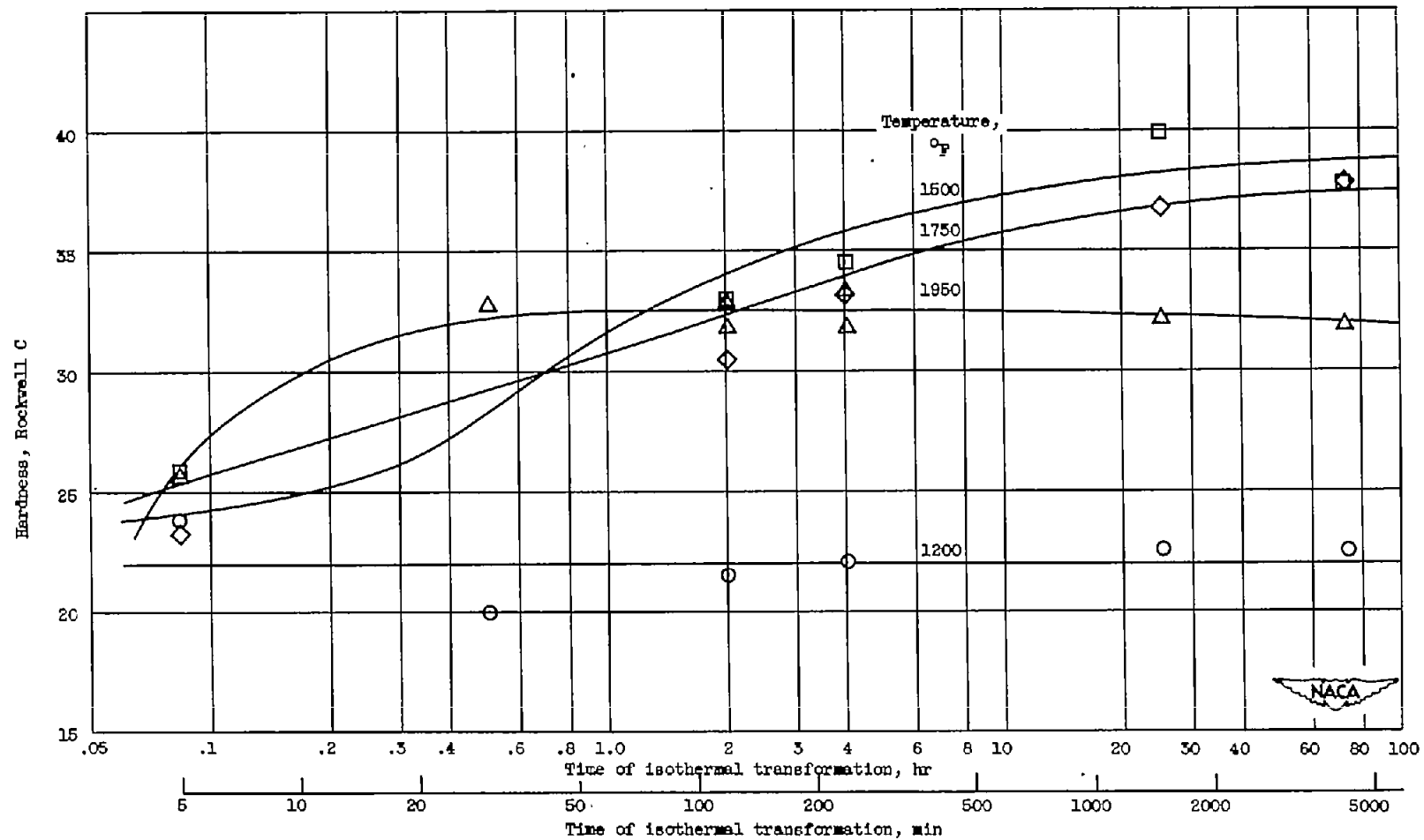


Figure 5. - Effect of time on hardness for isothermal transformation of solution-treated wrought Stellite 21 at various temperatures.

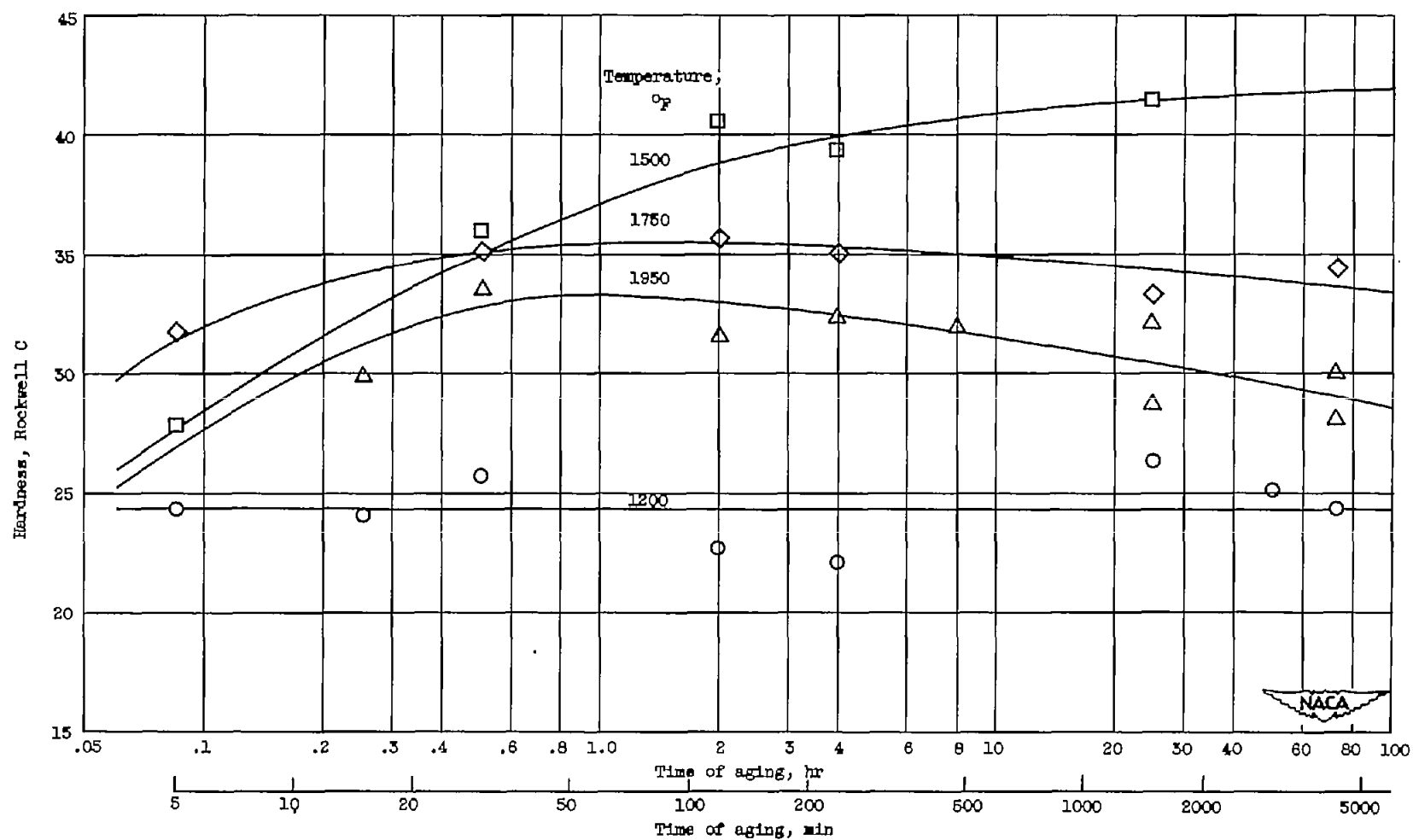


Figure 6. - Effect of time on hardness for aging solution-treated wrought Stellite 21 at various temperatures.

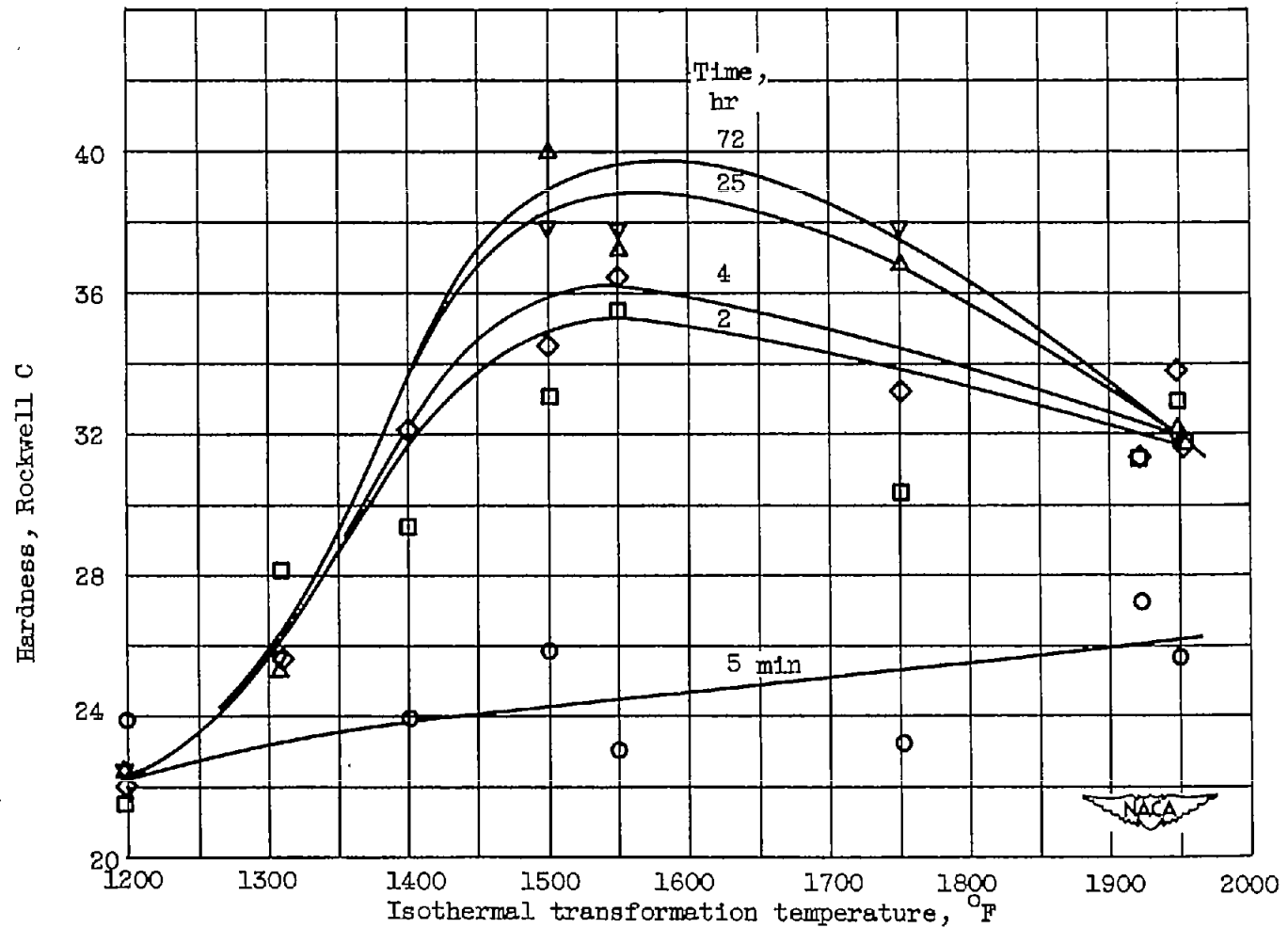


Figure 7. - Effect of temperature of isothermal transformation on hardness of wrought Stellite 21 for various times of isothermal transformation.

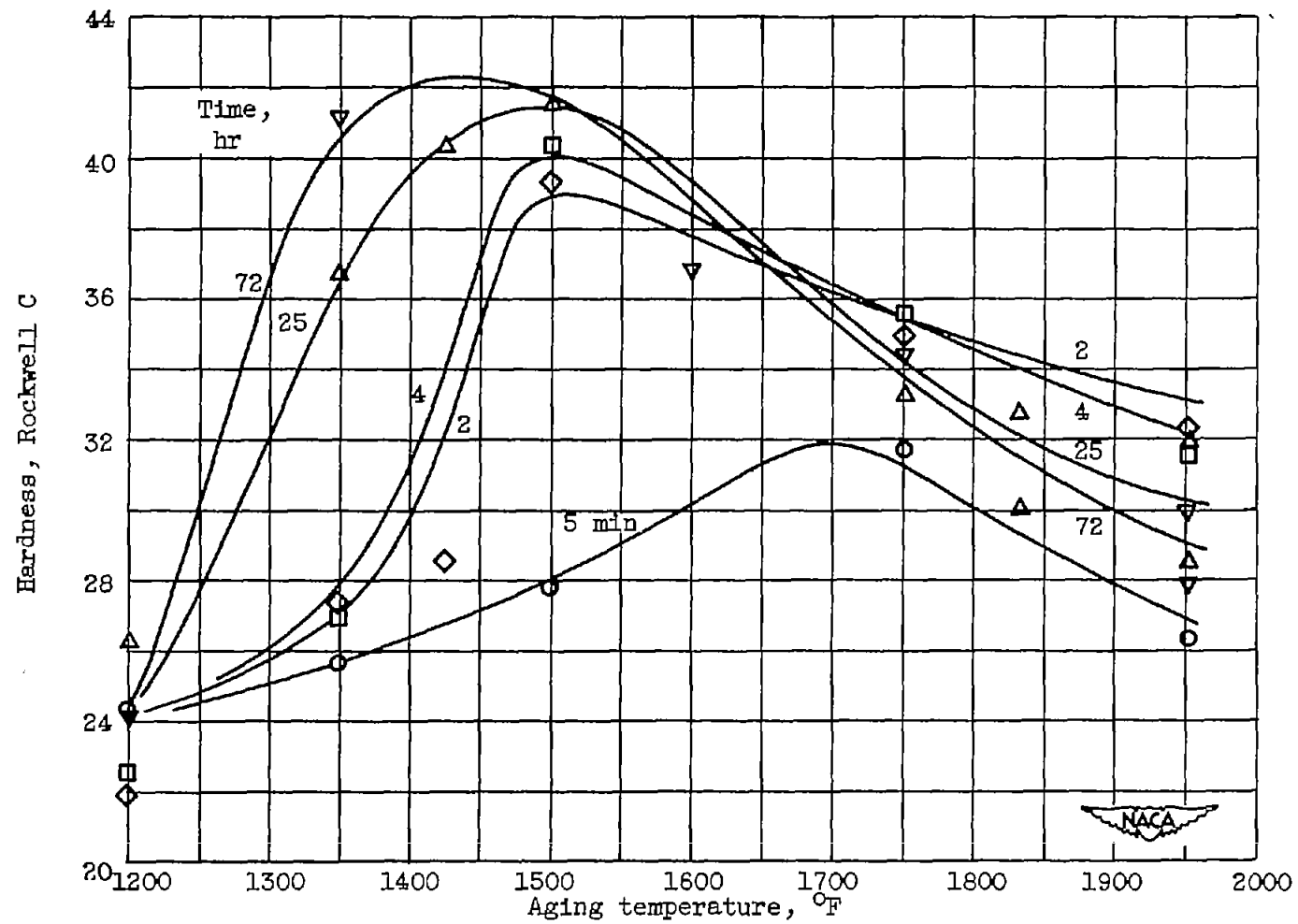
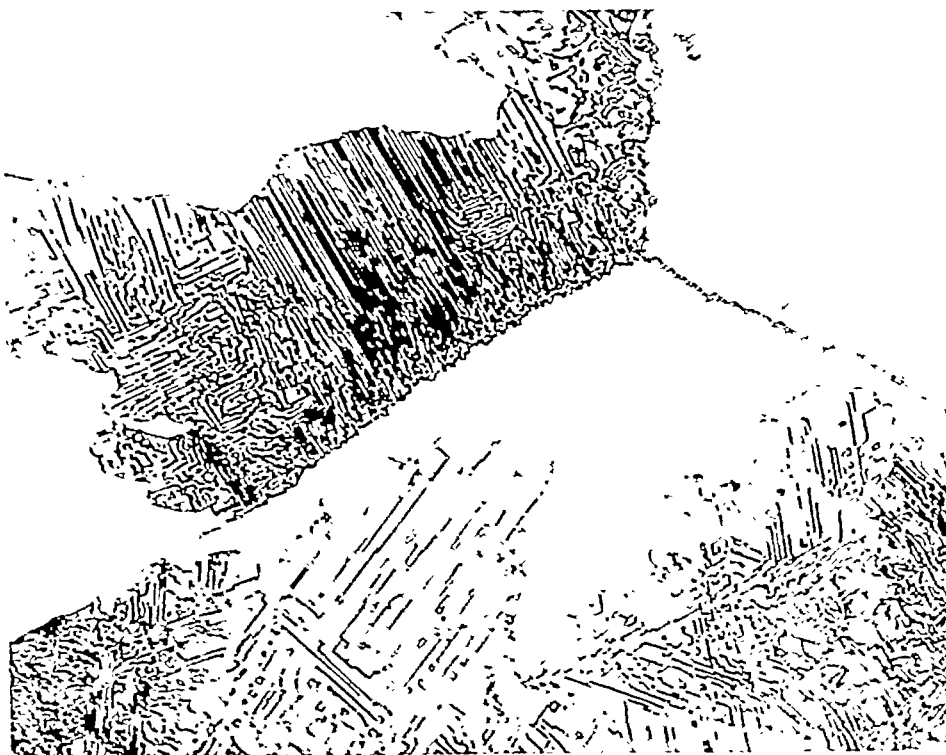


Figure 8. - Effect of aging temperature on hardness of wrought Stellite 21 for various times of aging.



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Figure 9. - Microstructure of heat-treated wrought Stellite 21 (16 hr at 2250° F, isothermally transformed 2 hr at 1950° F), showing pearlite surrounded by border. Electrolytically etched in 5 percent aqua regia. X750.